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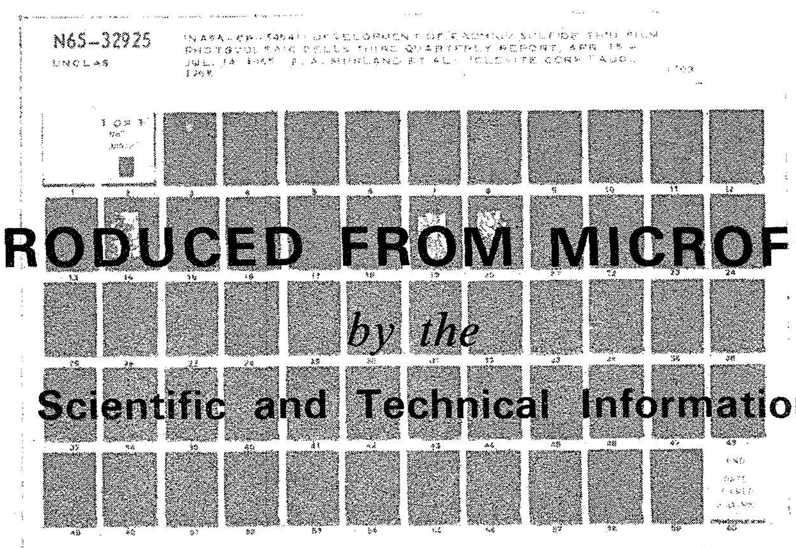

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Thermal Conductivity of Aerospace Alloys at Cryogenic Temperatures[1]

J. G. Hust, D. H. Weitzel and Robert L. Powell

Cryogenics Division
Institute for Materials Research
National Bureau of Standards
Boulder, Colorado 80302

An apparatus for measurement of thermal conductivity, electrical resistivity, and thermopower of metals and alloys is described. This apparatus, a modified version of the one used earlier in this laboratory, utilizes the steady-state, axial heat flow method. The samples are cylindrical rods about 23 cm long, and from 0.1 to 1.0 cm^2 in cross-sectional area. Included is a discussion about radiation induced errors, thermometry, and temperature control methods. Results on thermal conductivity, electrical resistivity, Lorenz ratio, and thermopower (with respect to normal-silver) are reported for titanium and aluminum alloys from 4° to 300°Kelvin. The data uncertainty is estimated to be about 1% below 120°K, but up to about 5% near 300°K when radiation effects become important.

Key Words Aluminum alloy, electrical conductivity, Lorenz ratio, thermal conductivity, thermopower, thermoelectric power, titanium alloy.

1. Introduction

The development of new materials for the aerospace industry has created a demand for data on the *thermal and electrical properties* of these alloys. To help satisfy the immediate needs for these data we are making thermal and electrical conductivity measurements from liquid helium temperatures to near room temperature on several alloys. Later more accurate measurements will be made on several materials to establish them as standard reference materials. Measured standard reference materials may be used to verify the accuracy of new thermal conductivity apparatus or as reference standards in comparative apparatus. The availability of measured standard reference materials should encourage more laboratories to enter this field of measurement.

Thermal data of technically important solids accurate to about 5% satisfy current demands. However, since future demands will likely become more stringent and because standard reference material data are also desired, this program is directed toward the acquisition of data which are accurate to within 1%. Thermal conductivity data accurate to within 1% are difficult to obtain for poor conducting alloys such as titanium A-110AT and especially difficult at temperatures above approximately 120°K.

This paper describes the method of measurement and the apparatus. *Included are data for titanium* A-110AT and aluminum 7039, along with a brief discussion of errors.

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2. Method of Measurement

Of the many methods described in the literature for the measurement of thermal conductivity, probably the simplest both conceptually and mechanically is the axial heat flow method. In this configuration the sample is in the form of a rod with constant cross-sectional area and the heat flow is one-dimensional along the axis of the rod. The defining equation for one-dimensional heat flow is

$$\dot{Q} = -\lambda A \frac{dT}{dX} \tag{1}$$

where $\dot{Q}$ is the rate of heat flow thru the rod; λ is thermal conductivity of the rod at temperature, T; A is cross-sectional area of rod; and dT/dX is the temperature gradient along the rod. The rate of heat flow, $\dot{Q}$, and area, A, are measured directly while the temperature gradient dT/dX can only be approximated from a finite number of measured values of T and X along the sample. For most apparatus there are only two or three points at which T and X are measured; in ours there are eight.

One can approximate the temperature gradient, dT/dX, by $\Delta T/\Delta X$. The quantity ΔT is the temperature difference between two adjacent measured points separated by a distance ΔX. This approximation of dT/dX has the advantage of simplicity but the disadvantage of being mathematically accurate only if the increments Δ ι are small or if λ is independent of temperature. However, if ΔT is too small, it will be inaccurate due to measurement errors. A second method of obtaining the temperature gradient is as follows. The T, X points are represented by a function $T = T(X)$ which in turn is differentiated to obtain the approximate temperature gradient, $T'(X) = dT/dX$. Caution must be exercised in choosing the function $T(X)$ and in determining its parameters to avoid introducing serious fitting errors, particularly in the slope.

3. Apparatus

The apparatus (Fig. 1) used in this experiment is essentially the same as that used earlier at this laboratory by R. L. Powell, et al [1].[2] The present system differs from the earlier ones principally by the addition of the floating sink and its automatic temperature controls. The floating sink allows greater flexibility in setting the temperature of the top of the sample. The temperature of the floating sink is automatically stabilized, thus greatly reducing drift of the sample temperature because of bath temperature drift. The temperature of the shell (0.5 mm thick stainless steel) surrounding the sample is automatically controlled to be the same as the temperature of the sample at the bottom thermocouple station. Three trim heaters spaced evenly along the shell enable one to match the shell and sample temperatures at those locations as well. Since the top of the shell is in good thermal contact with the sample thru the floating sink block, it is possible to closely match the shell temperature to the sample temperature at a total of five points. This adjustment reduces conduction losses thru the leads connected to the sample as well as thermal radiation losses. The measuring thermocouples and the shell-to-sample differential thermocouples (both Chromel vs Au + 0.07% Fe) are electrically insulated from the sample and the shell. A typical thermocouple mount is illustrated in figure 2. Thermal contact, while still maintaining electrical insulation, is obtained with epoxy cement and Apiezen N grease as shown in figure 2.

This apparatus is designed to measure also the thermopower and the electrical resistivity of the sample. The thermopower is determined by measuring the Seebeck voltage between the top and bottom thermocouple holders using 36 AWG "normal" silver wires. The absolute thermopower of normal silver has been determined; it is small compared to most metals and alloys. Electrical resistivity is determined by passing a known current through the sample (from the sample heater thru the sample to the system ground) and measuring the voltage drop across the sample between the top and bottom thermocouple mounts. The Seebeck voltage is subtracted from the total voltage drop to obtain the resistivity voltage drop.

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[2] Figures in brackets indicate the literature references at the end of this paper.

4. Samples

Two alloys were investigated: titanium alloy A-110AT (annealed) and aluminum alloy 7039-T61. The aluminum sample was ground into a cylinder with 0.1 cm^2 cross-sectional area and 23 cm length. It was supplied by ACF Industries, Inc., Albuquerque, New Mexico with a chemical analysis as follows:

Mn	Si	Ni	Cr	Cu	Fe	Ti	Be	Zn	Mg	Al
.23%	<.10	<.02	0.20	<.10	<.15	0.018	<.001	3.60	2.55	Balance

The heat treatment T-61 was in compliance with Kaiser Aluminum and Chemical Company proprietary heat treatment procedure. The titanium sample was ground into a cylinder of 1.0 cm^2 cross section and 23 cm length. It was supplied by Crucible Steel Company of America, Syracuse, New York with the following chemical analysis:

C	Fe	N	Al	H	Sn	Ti
0.07%	0.20	0.01	5.5	0.0158	2.5	Balance

The hardness and grain size of these samples will also be determined and reported in a later publication for more complete characterization of the samples.

5. Results and Discussion

The thermal conductivity, electrical resistivity, and thermopower of titanium alloy A-110AT and Al-7039 were measured from 5 to 300°K and their values are given in figures 3 thru 8. The Lorenz ratios were calculated from these data and are plotted in figures 9 and 10.

Since Ti A-110AT was the first sample investigated with this redesigned apparatus, more runs than normal were conducted to determine the precision and reproducibility of these measurements. The temperature ranges of these runs were adjusted to obtain considerable overlapping between runs not only for a given bath but also between baths, e.g., some runs using a L-He bath overlap runs using $L\text{-}H_2$ bath. The aluminum sample was measured over the same temperature range as the titanium sample but there was less overlap in each range and therefore fewer runs were required. The number of runs and temperature range investigated with the indicated baths are given below.

Bath	Number of Runs		Temperature range	
	Ti A-110AT	Al-7039	Ti A-110AT	Al-7039
LHe	7	4	5 - 32°K	5 - 28°K
LH_2	7	4	23 - 105°K	21 - 72°K
LN_2	10	6	66 - 150°K	72 - 210°K
CO_2 and Ethanol	3	2	199 - 236°K	198 - 225°K
Ice and Water	1	2	280 - 300°K	280 - 296°K

The scatter in the data for the individual runs and the deviation between overlapping runs are both less than 1% below 120°K. From 120 to 200°K slightly higher deviations are observed and are attributed to the presence of thermal radiation errors.

At the higher temperatures (between 200 and 300°K) the thermal conductivity curve as defined by each run contains a slight bump with the highest point corresponding to about the middle of the sample. This bump can be explained by considering the presence of a parallel path due to thermal radiation. *Due to the symmetry* of the sample and shield and the temperature distribution of each, the bottom of the sample (hot end) should experience a net loss of energy while the top should gain energy. Thus the calculated $\dot{Q}$ is more nearly correct nearest the heater, becomes progressively larger toward the middle of the sample, and becomes more nearly correct beyond the middle of the sample approaching the top end. Such an error in $\dot{Q}$ would tend to produce the observed bumps.

For Ti A-110AT the magnitudes of these "radiation" bumps are about 2% at 200°K and 6% at 300°K, while for Al-7039 the bump is masked by the scatter, i.e., less than 0.5%, at 200°K and is about 1% at 300°K. In an attempt to confirm that these bumps are caused by thermal radiation, a radiation shield composed of *loosely* packed *glass* wool was placed around the Al-7039 sample. An ice-bath run was repeated with this radiation shield in place and resulted in thermal conductivity values 6% lower than with no radiation shield. No bump is observed in these data. The measured thermal conductivity of Ti-A110AT at 300°K was lowered by 13% by the presence of the glass wool radiation shield. At 200°K the measured decrease was 6%, while at 115°K only a 1% decrease was observed. At 20°K the presence of the glass wool packing did not affect the measured value within the scatter of data. *It is concluded* that the size of the "bump" does not directly indicate the magnitude of the radiation errors, but the presence of the bump is direct evidence that radiation errors exist. The data presented in figures 3 thru 10 are consistent with the measurements made with the glass wool packing in the apparatus.

The present results have been computed with an estimated sensitivity curve for the Chromel vs Au + 0.07 atomic percent Fe thermocouples. This estimated curve may be systematically inaccurate by as much as 2% and adds directly to the uncertainty of the thermal conductivity data. The sensitivity of this thermocouple has been measured at this laboratory and the final data will soon be available. At that time thermal conductivity values will be recomputed using these more reliable data. The final computation will also be based on more refined data reduction techniques and will contain small corrections which have not been included yet. It is anticipated however that these changes will amount to no more than approximately 3%.

The details of these measurements, including a complete error analysis, will follow in a later publication.

6. References

[1] Powell, Robert L., Rogers, W. M., and Coffin, D. O., An Apparatus for Measurement of Thermal Conductivity of Solids at Low Temperatures, J. Res. NBS 59, No. 5, 349-55 (1957); RP 2805.

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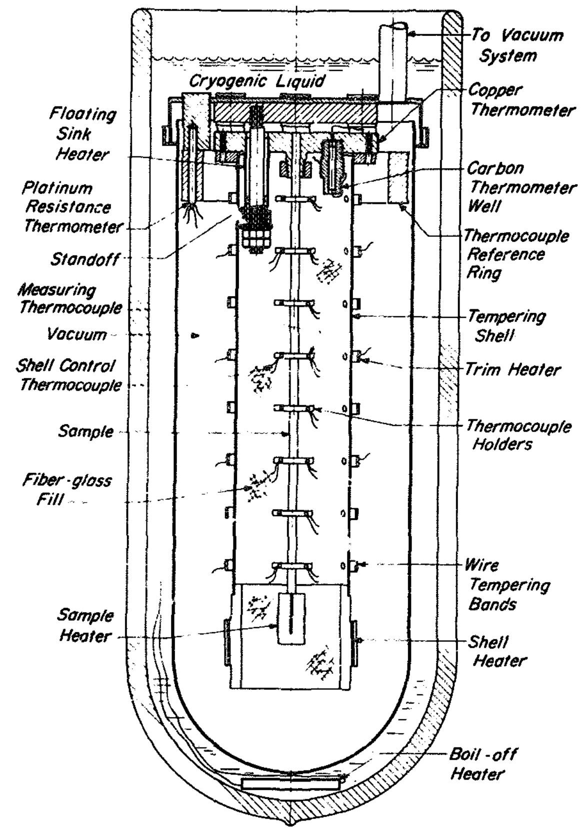

Figure 1. Thermal Conductivity Apparatus.

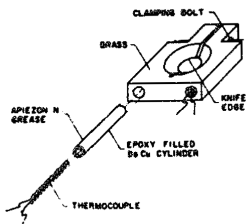

Figure 2. Thermocouple mount.

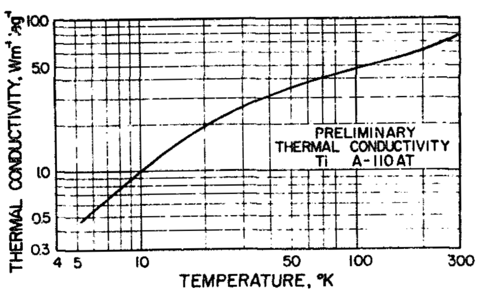

Figure 3. Thermal Conductivity of titanium alloy A-110AT.

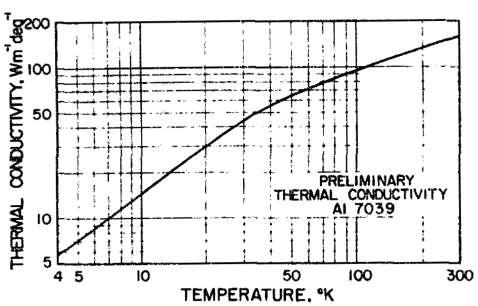

Figure 4. Thermal Conductivity of aluminum alloy 7039.

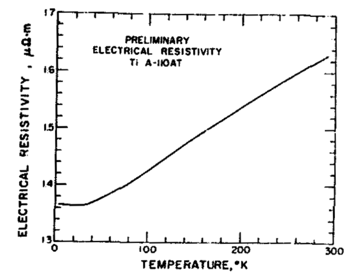

Figure 5. Electrical resistivity of titanium alloy A-110AT.

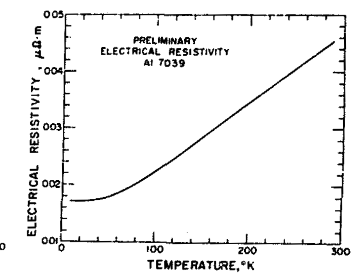

Figure 6. Electrical resistivity of aluminum alloy 7039.

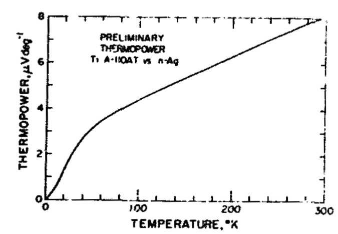

Figure 7. Thermopower of titanium alloy A-110AT. (vs. normal silver).

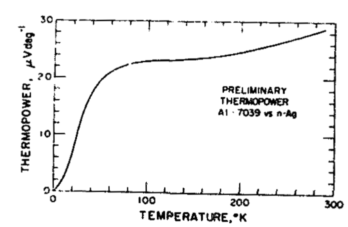

Figure 8. Thermopower of aluminum alloy 7039 (vs. normal silver).

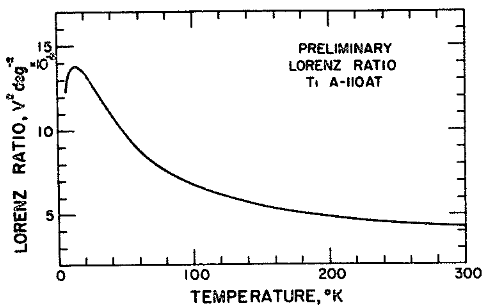

Figure 9. Lorenz ratio of titanium alloy A-110AT.

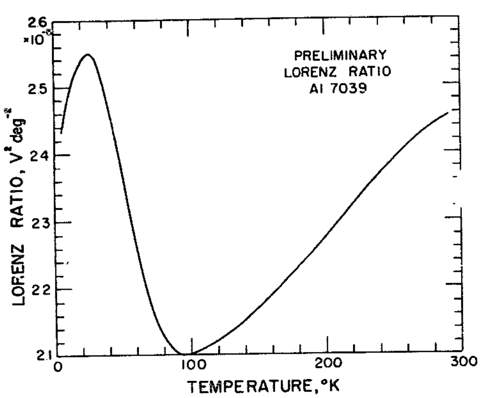

Figure 10. Lorenz ratio of aluminum alloy 7039.